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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460

NOV 2 1990

OFFICE OF  
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: PP# 9F3796 Sulfosate (Touchdown™) in or on corn grain, forage and fodder.

PP# 0F3860 Sulfosate (Touchdown™) in or on soybean seed, forage and hay.

Request for Petition Method Validation Trials of Analytical Methods WRC 85-33, WRC 85-34R, RRC 87-42 and RRC 87-41

MRID #: 412099-09,10,11,12,13,14,15,16,17,18,19  
412359-04  
412360-05  
414621-02,03,04,05,06

DEB#: N/A  
HED#: N/A

FROM: Steven R. Koepke, Ph.D., Chemist  
Tolerance Petition Section I  
Chemistry Branch I: Tolerance Support  
Health Effects Division (H7509C) *See 976*

THRU: Richard D. Schmitt, Ph.D., Chief  
Chemistry Branch I: Tolerance Support  
Health Effects Division (H7509C) *Richard D Schmitt*

TO: Donald A. Marlow, Chief  
Analytical Chemistry Branch  
Biological and Economics Analysis Division (H7503C)

ICI Americas Inc., Agricultural Products has proposed the establishment of tolerances for the combined residues of the herbicide sulfosate (Touchdown™), the trimethylsulfonium salt of N-phosphonomethylglycine (carboxymethylamino methyl phosphonate) and its metabolite, AMPA (aminomethylphosphonic acid) (calculated as the herbicide) in or on: corn grain @ 0.1 ppm, corn forage @ 0.2 ppm and corn fodder @ 0.2 ppm and soybean seed @ 2.0 ppm, soybean forage @ 1.0 ppm and soybean hay @ 3.0 ppm. Although ICI

has not proposed tolerances for animal commodities, such tolerances will be required.

Sulfosate is a NEW herbicide; there are no established tolerances for the trimethylsulfonium salt of glyphosate. Tolerances have been established for the isopropylamine (Roundup® herbicide) and sodium sesqui salts of glyphosate under 40 CFR 180.364. Sulfosate is similar in chemical structure, metabolic breakdown and proposed use to glyphosate.

The petitioner has submitted four primary proposed enforcement analytical methods, two each for the anion and cation of sulfosate. These are listed below:

For the cation:

**Analytical Method WRC 85-33**, "Determination of SC-0224 Cation Residues in Crops, Water and Soil by Gas Chromatography", Report No. WRC 85-33, June 15, 1989, by D.B. Katague and G.G. Patchett, MRID# 412359-04, 412099-17.

**Analytical Method RRC-87-42**, "Determination of Sulfosate Cation Residues in Milk, Eggs and Edible Tissues by Gas Chromatography", Method No. RRC-87-42, July 31, 1987, by T.J. Meyers, D.B. Katague, B.C. Buitrago and G.W. Schwab, MRID#'s 414621-05, 06.

For the anion:

**Analytical Method WRC 85-34R**, "Determination of SC-0224 Anion Residues in Crops, Water and Soil by Liquid Chromatography", Laboratory Project No. WRC 85-34R, June 15, 1989, by G.G. Patchett, D.B. Katague and B.C. Buitrago, MRID# 412360-05, 412099-17.

**Analytical Method RRC 87-41**, "Determination of Sulfosate Anion Residues in Milk, Eggs and Edible Tissues by Liquid Chromatography", Method No. RRC-87-41, July 31, 1987, by G.G. Patchett, B.C. Buitrago, D.J. O'Brien and G.W. Schwab, MRID# 414621-05, 06.

DEB requests that BEAD conduct a petition method validation trial on all four submitted analytical methods. Two detailed copies of four methods (containing representative chromatograms and recovery data) are appended hereto (Attachments 5, 6, 7, and 8).

Samples should be run in duplicate per the experimental design specified in Attachments 1, 2, 3 and 4. Please complete and return those Attachments as part of your report.

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Also, please include with your report, copies of the standard curves, sample calculations and representative chromatograms for controls and fortified samples. Any deficiencies in the methods, as written, should also be noted and reported.

We have confirmed with the Pesticide Repository (RTP; FTS 629-3951, Pat Beyer) that the necessary standards for the method validation trials are available from them.

The Registration Product Managers for sulfosate, Robert Taylor and Cynthia Giles, should be contacted directly concerning the priority for completion of these petition validation trials.

Please address your written reports to: Robert S. Quick, Section Head, Tolerance Petition Section I, Chemistry Branch I: Tolerance Support, Health Effects Division (H7509C).

Attachments (8):

1. Experimental design for conducting the petition method validation trial for sulfosate cation in crops.
2. Experimental design for conducting the petition method validation trial for sulfosate anion in crops.
3. Experimental design for conducting the petition method validation trial for sulfosate cation in milk and tissues.
4. Experimental design for conducting the petition method validation trial for sulfosate anion in milk and tissues.
5. Two detailed copies each of the analytical method (including chromatograms and recovery data) for the cation of sulfosate:

"Determination of SC-0224 Cation Residues in Crops, Water and Soil by Gas Chromatography", Report No. WRC 85-33, June 15, 1989, by D.B. Katague and G.G. Patchett, MRID# 412359-04, 412099-17.

6. Two detailed copies each of the analytical method (including chromatograms and recovery data) for the anion of sulfosate:

"Determination of SC-0224 Anion Residues in Crops, Water and Soil by Liquid Chromatography", Laboratory Project No. WRC 85-34R, June 15, 1989, by G.G. Patchett, D.B.

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Katague and B.C. Buitrago, MRID# 412360-05,  
412099-17.

7. Two detailed copies each of the analytical method (including chromatograms and recovery data) for the cation of sulfosate:

"Determination of Sulfosate Cation Residues in Milk, Eggs and Edible Tissues by Gas Chromatography", Method No. RRC-87-42, July 31, 1987, by T.J. Meyers, D.B. Katague, B.C. Buitrago and G.W. Schwab, MRID#'s 414621-05 and 06.

8. Two detailed copies each of the analytical method (including chromatograms and recovery data) for the anion of sulfosate:

"Determination of Sulfosate Anion Residues in Milk, Eggs and Edible Tissues by Liquid Chromatography", Method No. RRC-87-41, July 31, 1987, by G.G. Patchett, B.C. Buitrago, D.J. O'Brien and G.W. Schwab, MRID# 414621-05 and 06.

cc (with all attachments):

P. Corneliussen (FDA, HFF-426)  
R. Ellis (USDA, FSIS)

cc (with Attachments 1, 2, 3 and 4 only):

S. Koepke (DEB)  
PP#9F3796  
PP#0F3860  
R. Taylor/C. Giles (PM#25/RD)  
C. Furlow (PIB/FOB)  
E. Greer Jr. (ACB/BEAD)  
R. Thompson (RTP-NC)  
Circulation(7)  
RF  
SF

H7509C:DEB:Reviewer(SK):CM#2:Rm812A:557-4380:Typist(SK):  
10/26/90.

RDI:Section Head: R.S. Quick:10/26/90: Br.Sr.Scientist:R.A.  
Loranger: 10/30/90.

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ATTACHMENT 1

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METHOD: "Determination of SC-0224 Cation Residues in Crops, Water and Soil by Gas Chromatography", Report No. WRC 85-33, June 15, 1989, by D.B. Katague and G.G. Patchett, MRID# 412359-04, 412099-17.

Please: (1) Indicate the limit of detection; (2) Do Not use control values for recovery calculations; and (3) Do Not report control values as zero; if less than the limit of detection, report as such.

<u>Commodity</u>	<u>Chemical Added*</u>	<u>PPM added</u>	<u>PPM found</u>	<u>% Recovery</u>
Corn Grain	TMS	0.0		
	"	0.05		
	"	0.1		
<hr/>				
Corn Forage	TMS	0.0		
	"	0.1		
	"	0.2		

\* TMS = Trimethylsulfonium Chloride

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ATTACHMENT 1

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Modifications made to Method:

Special precautions to be taken:

Source of analytical reference standards:

If derivatized standard used, give source:

Instrumentation for quantitation:

Instrumentation for confirmation:

If instrumentation parameters differ from those in the Method,  
list parameters actually used:

Commercial source for any special chemicals or apparatus:

Comments:

Chromatograms:

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ATTACHMENT 2

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METHOD: "Determination of SC-0224 Anion Residues in Crops, Water and Soil by Liquid Chromatography", Laboratory Project No. WRC 85-34R, June 15, 1989, by G.G. Patchett, D.B. Katague and B.C. Buitrago, MRID# 412360-05, 412099-17.

Please: (1) Indicate the limit of detection; (2) Do Not use control values for recovery calculations; and (3) Do Not report control values as zero; if less than the limit of detection, report as such.

<u>Commodity</u>	<u>Chemical Added*</u>	<u>PPM added</u>	<u>PPM found</u>	<u>% Recovery</u>
Corn Grain	CMP	0.0		
	"	0.05		
	"	0.1		
	AMPA	0.0		
	"	0.05		
	"	0.1		
Corn Forage	CMP	0.0		
	"	0.1		
	"	0.2		
	AMPA	0.0		
	"	0.1		
	"	0.2		

\* CMP = carboxymethylamino methyl phosphonate  
 AMPA = aminomethylphosphonic acid

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ATTACHMENT 2

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Modifications made to Method:

Special precautions to be taken:

Source of analytical reference standards:

If derivatized standard used, give source:

Instrumentation for quantitation:

Instrumentation for confirmation:

If instrumentation parameters differ from those in the Method,  
list parameters actually used:

Commercial source for any special chemicals or apparatus:

Comments:

Chromatograms:

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ATTACHMENT 3

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METHOD: "Determination of Sulfosate Cation Residues in Milk, Eggs and Edible Tissues by Gas Chromatography", Method No. RRC-87-42, July 31, 1987, by T.J. Meyers, D.B. Katague, B.C. Buitrago and G.W. Schwab, MRID#'s 414621-05, 06.

Please: (1) Indicate the limit of detection; (2) Do Not use control values for recovery calculations; and (3) Do Not report control values as zero; if less than the limit of detection, report as such.

<u>Commodity</u>	<u>Chemical Added*</u>	<u>PPM added</u>	<u>PPM found</u>	<u>% Recovery</u>
Milk	TMS	0.0		
	"	0.02		
	"	0.05		
Cow Liver	TMS	0.0		
	"	0.1		
	"	0.2		

\* TMS = Trimethylsulfonium Chloride

ATTACHMENT 3

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Modifications made to Method:

Special precautions to be taken:

Source of analytical reference standards:

If derivatized standard used, give source:

Instrumentation for quantitation:

Instrumentation for confirmation:

If instrumentation parameters differ from those in the Method, list parameters actually used:

Commercial source for any special chemicals or apparatus:

Comments:

Chromatograms:

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ATTACHMENT 4

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METHOD: "Determination of Sulfosate Anion Residues in Milk, Eggs and Edible Tissues by Liquid Chromatography", Method No. RRC-87-41, July 31, 1987, by G.G. Patchett, B.C. Buitrago, D.J. O'Brien and G.W.Schwab, MRID# 414621-05, 06.

Please: (1) Indicate the limit of detection; (2) Do Not use control values for recovery calculations; and (3) Do Not report control values as zero; if less than the limit of detection, report as such.

<u>Commodity</u>	<u>Chemical Added*</u>	<u>PPM added</u>	<u>PPM found</u>	<u>% Recovery</u>
Milk	CMP	0.0		
	"	0.02		
	"	0.05		
	AMPA	0.0		
	"	0.02		
	"	0.05		
Cow Liver	CMP	0.0		
	"	0.2		
	"	0.5		
	AMPA	0.0		
	"	0.2		
	"	0.5		

\* CMP = carboxymethylamino methyl phosphonate  
 AMPA = aminomethylphosphonic acid

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ATTACHMENT 4

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Modifications made to Method:

Special precautions to be taken:

Source of analytical reference standards:

If derivatized standard used, give source:

Instrumentation for quantitation:

Instrumentation for confirmation:

If instrumentation parameters differ from those in the Method, list parameters actually used:

Commercial source for any special chemicals or apparatus:

Comments:

Chromatograms:

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